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LLNL-TR-701561

Compilation of LLNL CUP-2 Data

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August 26, 2016

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This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

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Lawrence Livermore National Laboratory
Nuclear Forensics Group

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SAMPLE BACKGROUND

The CUP-2 uranium ore concentrate (UOC) standard reference material, a powder, was produced at the Blind River uranium refinery of Eldorado Resources Ltd. in Canada in 1986. This material was produced as part of a joint effort by the Canadian Certified Reference Materials Project and the Canadian Uranium Producers Metallurgical Committee to develop a certified reference material for uranium concentration and the concentration of several impurity constituents. This standard was developed to satisfy the requirements of the UOC mining and milling industry, and was characterized with this purpose in mind.

To produce CUP-2, approximately 25 kg of UOC derived from the Blind River uranium refinery was blended, homogenized, and assessed for homogeneity by X-ray fluorescence (XRF) analysis. The homogenized material was then packaged into bottles, containing 50 g of material each, and distributed for analysis to laboratories in 1986. The CUP-2 UOC standard was characterized by an interlaboratory analysis program involving eight member laboratories, six commercial laboratories, and three additional volunteer laboratories. Each laboratory provided five replicate results on up to 17 analytes, including total uranium concentration, and moisture content. The selection of analytical technique was left to each participating laboratory. Uranium was reported on an "as-received" basis; all other analytes (besides moisture content) were reported on a "dry-weight" basis. Certified concentrations of the CUP-2 standard can be found in Table 1, and a copy of the CUP-2 UOC certificate can be found in Annex 1. A photograph of the sample bottle can be seen on the cover of this report.

Constituent	Concentration (wt. %)	Uncertainty
$U^{\S}$	75.42	0.17
Moisture	2.94	-
S	0.80	-
Ca	0.62	-
Na	0.459	-
Fe	0.311	-
Mg	0.229	-
Si^*	0.17	-
K^*	0.11	-
Mo	0.069	-
V^*	0.066	-
Zr^*	0.044	-
As	0.035	-
P^*	0.030	-
Ti	0.019	-
B^*	0.0051	-
Ni	0.0029	-

Table 1. Concentration of major and trace element constituents in CUP-2 UOC standard reference material, reported on an "as-received" basis, per the certificate of analysis provided by the manufacturer. §, reported on a dry weight basis; *, provisionally recommended concentration. Uncertainty is only provided for the U concentration measurement.

A bottle of 25g of CUP-2 UOC standard as described above was purchased by LLNL and characterized by the LLNL Nuclear Forensics Group. The non-destructive and destructive analytical techniques listed in Table 2 were applied to the UOC sample. The analytical sequence was structured so that non-destructive, short-term analyses were performed before longer-term, destructive analyses. Information obtained from short-term techniques such as photography, gamma spectrometry, and scanning electron microscopy were used to guide the performance of longer-term techniques such as ICP-MS. Some techniques, such as XRF and ICP-MS, provided complementary types of data: in this case, the shorter-term technique (XRF) was initially utilized in data interpretation, until more precise and accurate (ICP-MS) data were used for later interpretations. Only about 2 g of the 25 g present in the bottle was required for use in this study.

Technique applied	**Type of technique**	**Type of information**
Photography	Non-destructive	Initial characterization
Dosimetry	Non-destructive	Dose rate
Gamma-spectrometry	Non-destructive*	Activity of short-lived nuclides
Scanning electron microscopy	Non-destructive*	Spatially-resolved textural and compositional variation
X-ray diffraction	Non-destructive*	Presence and abundance of crystalline phases
X-ray fluorescence	Non-destructive*	Concentration of trace elements present in concentrations > 100 ppm
U assay	Destructive	Concentration of uranium
Inductively-coupled mass spectrometry	Destructive	Concentration of trace elements present in concentrations > 10 ppt, isotopic composition of uranium

Table 2. List of analytical techniques utilized in CUP-2 UOC analyses.

PHOTOGRAPHY

The CUP-2 powder was initially photographed in the bottle using a Canon EOS Rebel T3i single-lens reflex digital camera, with zoom lens. The bottle was opened, and the camera was pointed directly down the opening of the bottle, in order to visualize the top layer of UOC powder in the bottle. From the photograph (Figure 1), the sample appeared to be a finely-grained, clumpy orange-red powder. Small specks of a slightly lighter material can be visualized in the powder, distributed roughly evenly throughout. It is unclear through the photograph alone if these lighter specks are a different material than the bulk of the rest of the powder. The clumpy nature of the powder suggests that it may contain some moisture.

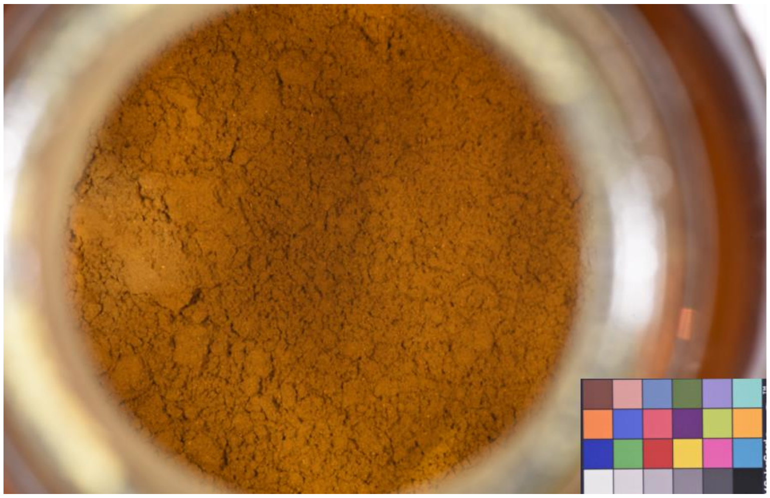


Figure 1. Digital photograph of CUP-2 powder. The photograph was taken with the digital camera aiming down the opening of the bottle. A color palette is included in the lower right of the photograph, for reference.

GAMMA SPECTROMETRY

2.0362 g of CUP-2 powder and a solution of 26.6667 mL dissolved CUP-2 powder were each counted in two different configurations with a Canberra Broad Energy Ge detector. For calculation of the U isotopic composition using the spectral analysis program MGAU 4.2, the samples were counted for at least 100 minutes flush against the detector face, over 4096 channels, and gains set to give 0.075 keV per channel. To check the performance of MGAU with this detector, two U standard solutions (one depleted and one low-enriched) were also counted, and the results are given in Table 3. The MGAU results are accurate enough for basic categorization, but are biased well beyond the stated uncertainties for the LEU case. A possible reason for sub-optimal results is the limited resolution of the BEGe detector (which is 0.3 cm thick with a 69 mm diameter), such analysis is best performed on a thin low-energy detector. However, this program is useful for calculating the isotopic composition immediately, and appears to be accurate to within +/- 0.3% weight.

	MGAU (wt%)	U050 MGAU +/-	NBS Provisional Certificate wt%	NBS +/-	MGAU bias	Bias/σ
U050						
U-234	0.0301	0.0006	0.02745	0.0001	0.00265	4.41666667
U-235	4.7602	0.0319	4.95	0.005	-0.1898	-5.94984326
U-236	Not calculated		0.0476	0.0002	N/A	
U-238	95.2097	0.0323	94.975	0.005	0.2347	7.26625387
U0002						
U-234	0	0.0003	0.000157	0.000001	-0.000157	-0.52333333
U-235	0.0007	0.0813	0.01733	0.00005	-0.01663	-0.20455105
U-236	Not calculated					
U-238	99.9993	0.0823	99.983	0.005	0.0163	0.19805589

Table 3. Results of gamma spectrometry analyses of calibration solutions U-050 and U-0002.

Table 4 shows that the MGAU analysis of the powder and solution samples revealed the CUP-2 powder has a natural isotopic composition. Furthermore, there is no significant difference if the sample is measured as a loose powder, or as dissolved in solution. The difference in ^{234}U abundance reported for the powder and solution sample is not significant; it is well known that MGAU analysis is less accurate for ^{234}U abundance than for ^{235}U abundance. An image of the gamma spectrum of the dissolved CUP-2 solution can be seen in Figure 2.

	Powder		**Solution**	
Isotope	Abundance (wt%)	Uncertainty	Abundance (wt%)	Uncertainty
U-234	0.0053	0.0005	0.0069	0.0012
U-235	0.700	0.015	0.710	0.044
U-238	99.294	0.015	99.283	0.044

Table 4. Results of gamma spectrometry analyses of CUP-2 UOC powder and dissolved solutions.

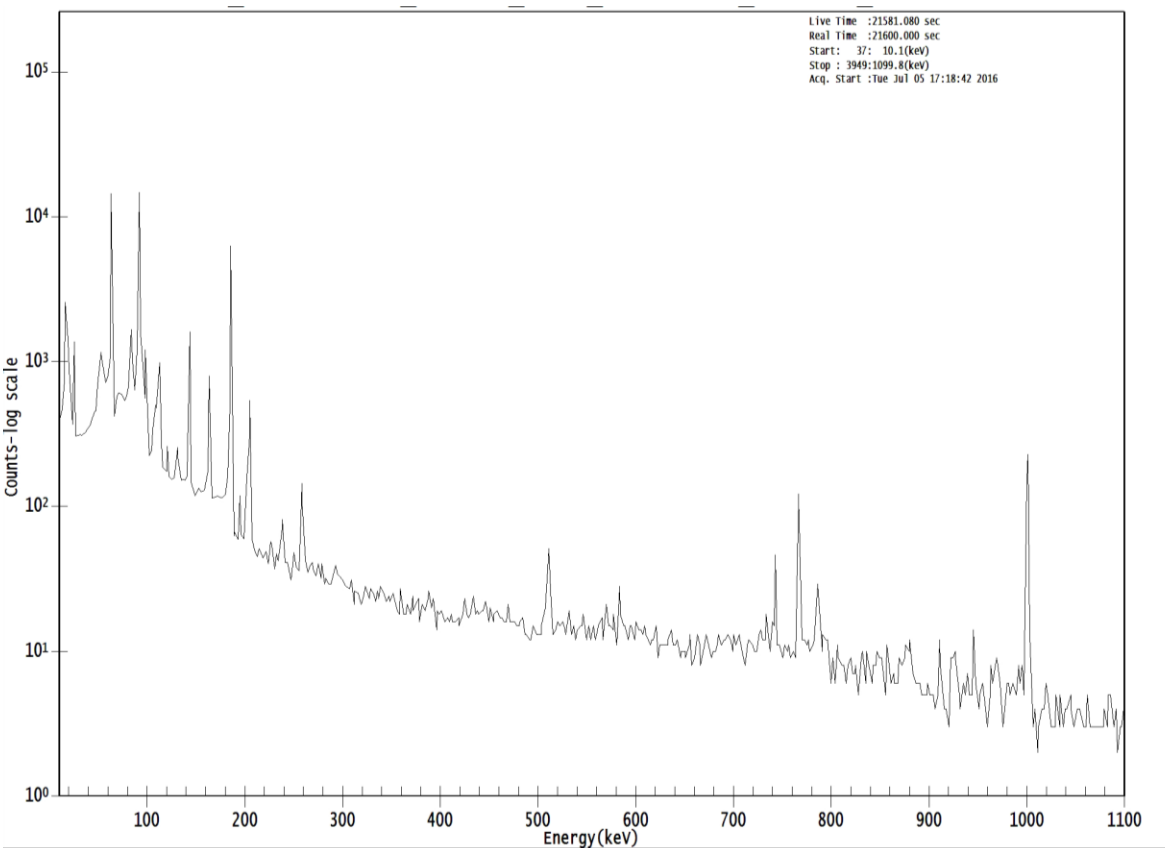


Figure 2. Gamma spectrum of CUP-2 UOC dissolved in solution.

The samples were also counted for 6 hours in a position 7.7 cm away from the detector, with gains covering the energy region 10-2000 keV, in order to detect any radioactive contaminants and daughter products. Only the immediate daughters of U (^{231}Th, ^{234}Th, ^{234m}Pa) were detected, suggesting that the sample is not old enough for γ detection of ^{231}Pa or ^{226}Ra decay chain products. The radioactive impurities detected were ^{228}Th daughters. When ^{228}Th daughters are detected without peaks from ^{228}Ac this can indicate the presence of ^{232}U. However, these peaks could also be an artifact of natural background fluctuations, and are not significant enough in the CUP-2 spectrum to assert the presence of ^{232}U. No qualitative differences in the gamma spectra were observed between the powder and aqueous samples. Quantitative results are reported for the aqueous sample in Table 5. These results should be more accurate than those from the powder sample because in the case of the aqueous sample, the efficiency curve could be obtained with a geometry-matched standard. The ^{235}U enrichment calculated from the peak fitting approach yields a consistent result with the MGAU analysis, concluding that the composition is natural. The ^{234}U abundance is not consistent, and is unrealistically low the natural composition. The value from the peak fit has high uncertainty, and is also likely underestimated due to the large Compton continuum in the low energy region of the spectrum.

Nuclide	**Bq**	**±2σ**	**Atoms**	**±2σ**
Th-228	0.29	0.11	2.51E+07	9.6E+06
Th-231	52.4	14	1.68E+18 (U-235)	4.6E+17
Pa-234m	943	94	1.92E+20 (U-238)*	1.9E+19
Th-234	939	206	1.91E+20 (U-238)*	4.2E+19
U-234	1146	361	1.3E+16	4.0E+15
U-235	42.8	3.0	1.370E+18	9.6E+16

Table 5. Radionuclides detected in CUP-2 dissolved sample via gamma spectrometry. *Calculation of ^{238}U atoms assumes secular equilibrium (no chemical separations 5 months prior to counting)

SCANNING ELECTRON MICROSCOPY – ENERGY DISPERSIVE SPECTROSCOPY

Samples of the CUP-2 UOC standard were imaged by scanning electron microscope (SEM) using the FEI Inspect F SEM at LLNL to characterize both the morphology and elemental composition of the powder. Sample preparation for SEM analysis involved depositing small amounts of the CUP-2 material onto carbon sticky tape.

The SEM instrument used at LLNL is equipped with a high brightness Schottky field emission gun (FEG) which produces images with a spatial resolution of up to 1.2 nm at 30 kV. Secondary electron (SE) and backscattered electron (BE) images were collected to characterize both the morphology and atomic number (Z) contrast. Energy dispersive X-ray (EDX) measurements were carried out using a Bruker Si(Li) detector at 20 kV and 10 mm working distance. Images were collected using 5 kV accelerating voltage for imaging and 20 kV for energy dispersive X-ray analysis.

Description of Particle Agglomerates

In Figure 3, several large agglomerates of powder, varying from ~200 to 500 µm, can be seen. Since the material was poured directly onto sticky carbon tape, it is clear that the particles of this material tend to stick together in clumps. In Panel A, several agglomerates of the powder can be observed, in differing sizes. Even in this large field of view, it is clear that this material is heterogeneous in grain size, and perhaps in morphology. Each agglomerate of material seems to consist of hundreds to thousands of individual particles. Panel B depicts a zoomed-in view of an agglomerate of about 200 µm in diameter. Heterogeneity in grain size is clearly observed, with the largest particles extending to almost 40 µm in diameter, and with smaller resolvable particles on the scale of several µm. Most of the observable particles in this panel have high sphericity, but have some degree of angularity. A few rod-like or acicular particles can be identified, but are a minor component here. In Panel C, the secondary electron image of a different agglomerate is depicted. This agglomerate seems to contain a smaller proportion of the largest sub-spherical particles, and instead contains a larger fraction of sub-micron, acicular particles. The acicular particles tend to be intermingled with the sub-spherical particles. Panel D depicts a third, much larger (~500 µm in diameter) agglomerate of material. The largest particles in this agglomerate approach 75 µm in diameter, and this agglomerate tends to be dominated by the sub-spherical particles.

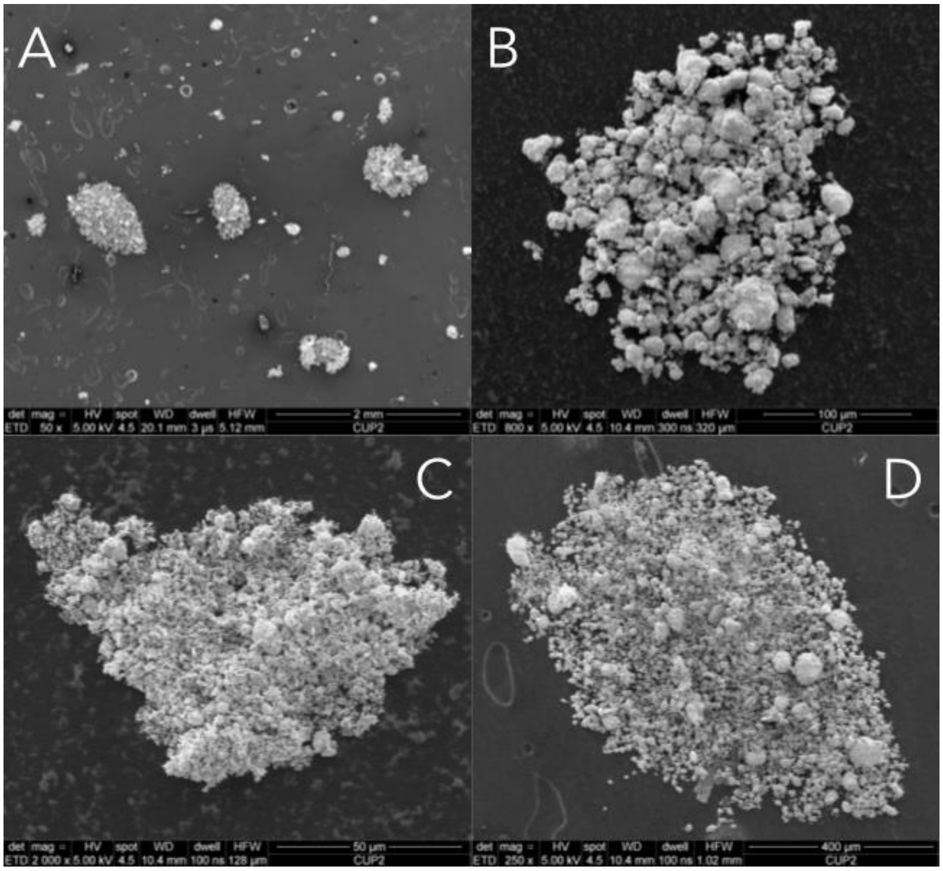


Figure 3. Secondary electron images of CUP-2 material showing large agglomerates on carbon sticky tape.

Description of Particles

Figure 4 depicts a closer-in view of the different types of particles observed in this material. Panel A is dominated by the sub-spherical particles, of various sizes. Some of these particles appear to be agglomerations of smaller sub-spherical particles, containing a fraction of the acicular particles. In addition, several large, plate-like particles are observed. These particles appear to be thicker than the acicular ones, and they appear to have a fine-grained coating on their surface. Panel B depicts a closer-up view of many of the sub-spherical particles, averaging ~25 µm in diameter. In this image, it is clear that many of these particles contain a fine-grained coating, which in some cases looks like it is binding individual larger particles together. This can also be seen in Panel C. In addition, larger (~25 µm) diameter agglomerations of the acicular particles can be observed in the left portion of the panel. These appear to be different from the sub-spherical particles that proportionally dominate the panel. Panel D depicts a portion of the material dominated by the finest-grained acicular particles.

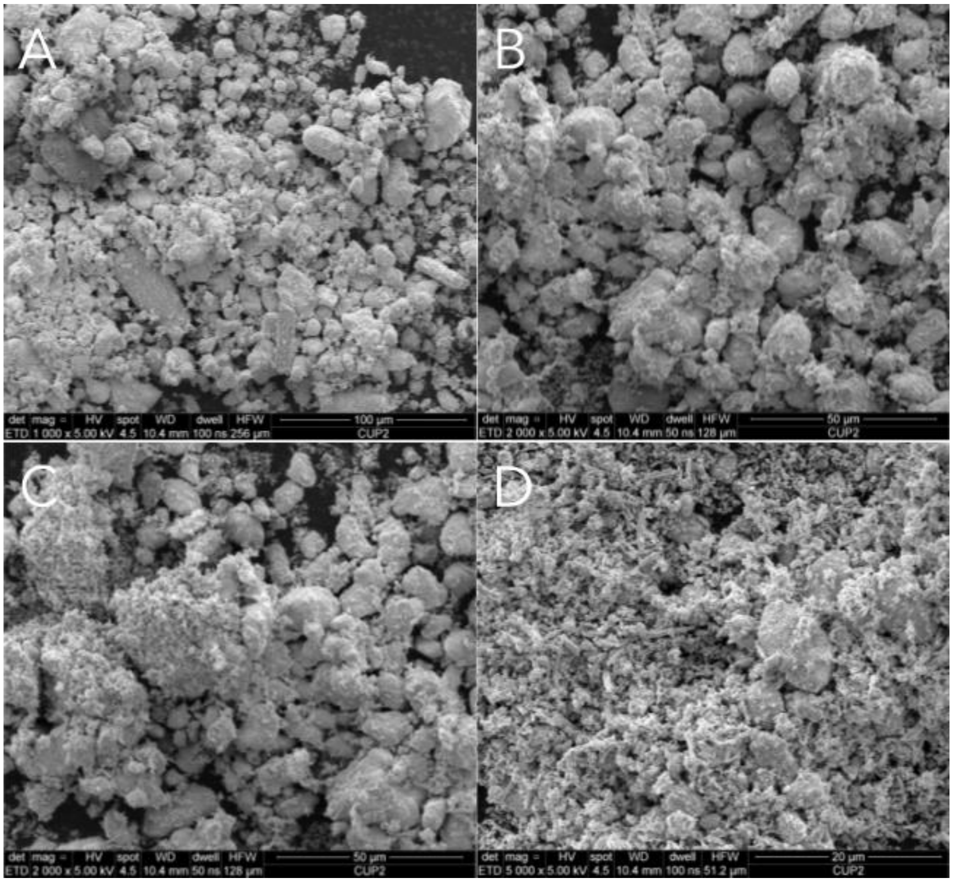


Figure 4. Secondary electron images of CUP-2 material showing different types of particles.

Description of Particle Texture

Figure 5 depicts the textures of individual particles. In Panel A, several sub-spherical particles are shown. On the surface, they contain a significant fraction of sub-µm acicular material as a coating. In some places, the acicular grains form agglomerates independently of the sub-spherical particles. Panel B depicts a region of material consisting almost entirely of the acicular particles, with perhaps a minor component of the smallest fraction of the sub-spherical particles. In Panel C, a single ~30 µm sub-spherical particle, with its acicular coating, is observed. Although it is difficult to determine the exact shape of the sub-spherical particle, it appears to be relatively well-rounded. Panel D depicts a mixture of both sub-spherical and acicular particles. Again, many of the sub-spherical particles have a significant coating of the finest-grained acicular material, which may work to bind the sub-spherical particles together.

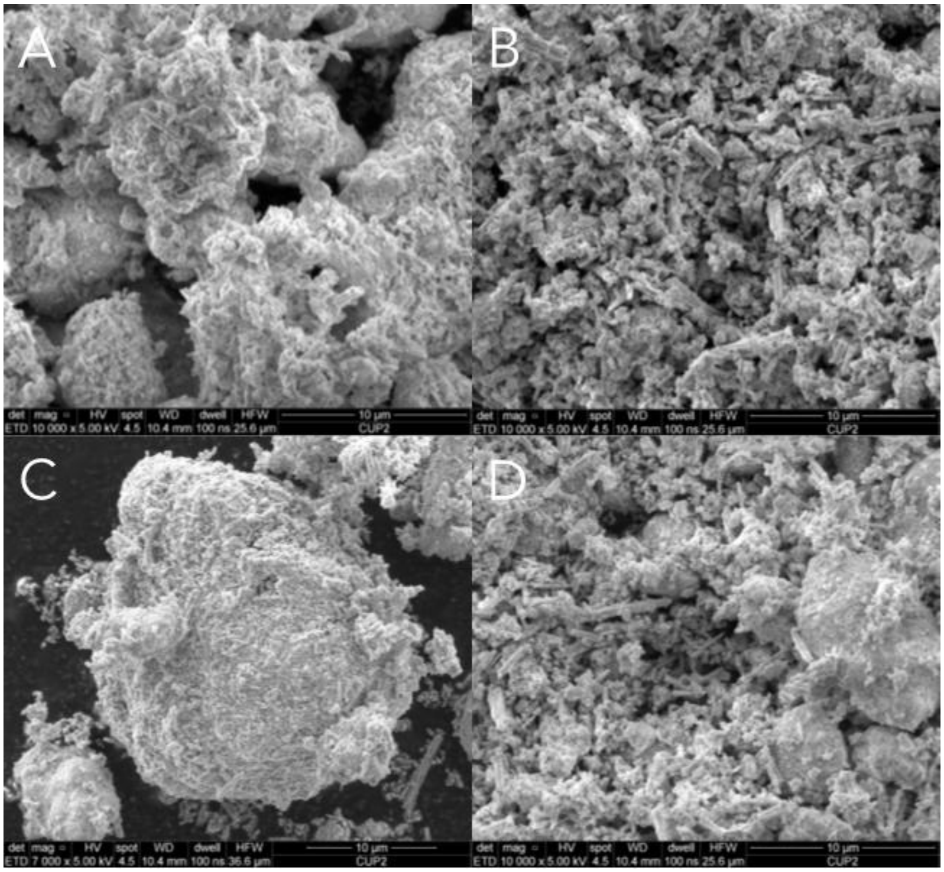


Figure 5. Secondary electron images of CUP-2 material showing particle texture.

<u>High magnification SEM imaging</u>

Figure 6 shows some high magnification SEM images of the CUP-2 material. In Panel A, individual particles of the acicular material can be clearly observed. These particles are sub-µm in the short dimension. In addition, a small fraction of the smallest sub-spherical particles can be observed in the upper right portion of the panel. In Panel B, additional acicular particles are observed. Some contain a few particles of an even smaller-grained material. Panel C depicts the acicular coating of a larger sub-spherical particle. This coating has a relatively homogeneous particle size, and the acicular rods tend to be shorter in the longest dimension than the acicular particles found in other locations. Panel D clearly depicts one of the plate-like particles. These particles make up only a small fraction of the total material, but tend to be fairly notable in appearance. The surface of this particle is smoother than the surface of the sub-spherical particles, and contains a minor coating of the acicular material. There appears to be striations on the surface of this particle, oriented laterally with respect to overall dimension. It is unclear if these striations are due to the coating, or whether they are features present in the plate-like particle.

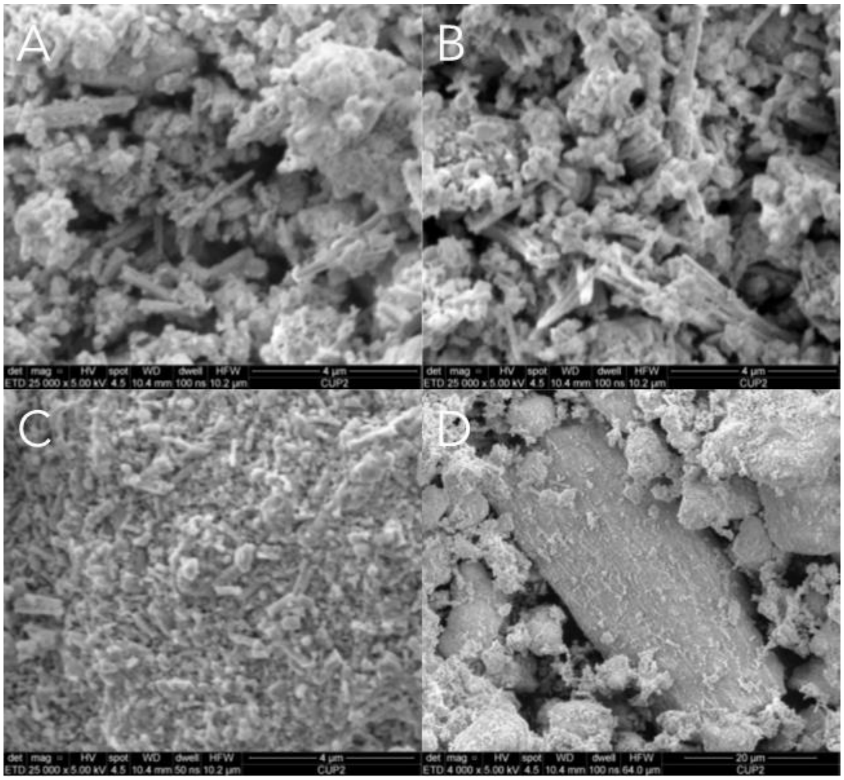
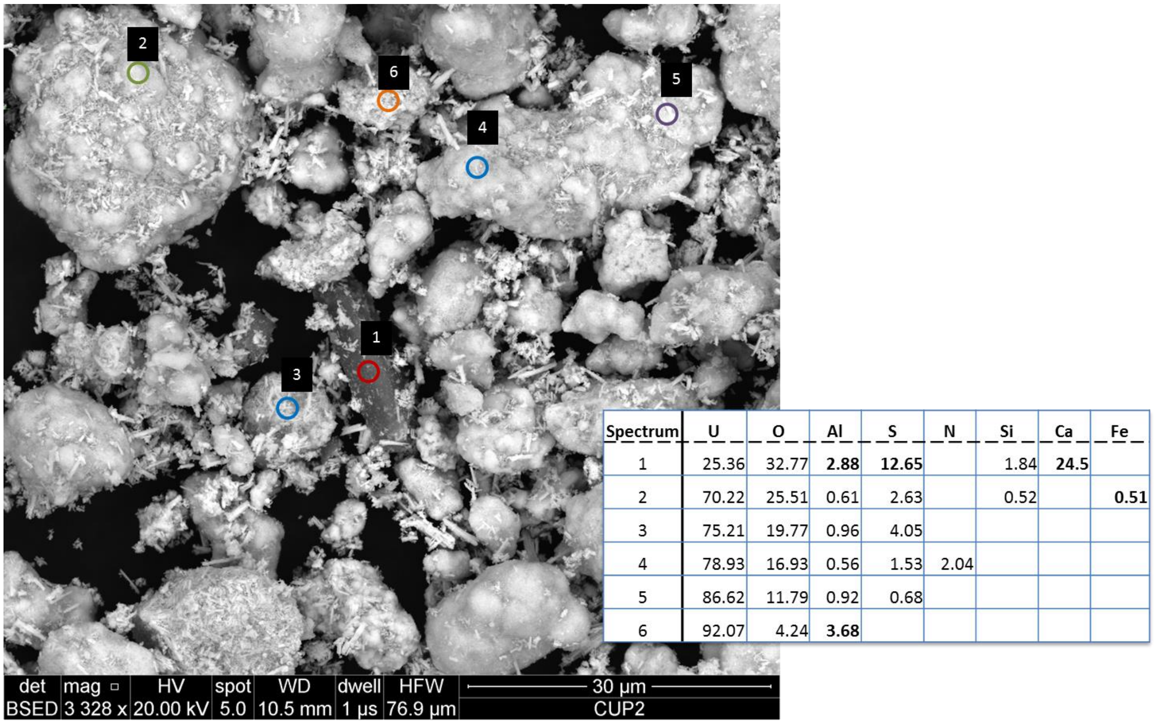


Figure 6. High magnification secondary electron images of CUP-2 material.

Energy-dispersive X-ray spectrometry

Energy-dispersive X-ray spectrometry was performed using the SEM-EDX system to characterize the chemical composition of the different particle types observed in the secondary electron images. With the measurement parameters applied for this sample, the analysis volume (interaction volume between sample and electron beam) was around 1 µm in diameter.

In Figure 7, backscattered SEM images are presented, with EDX data collection regions identified. Individual sub-spherical particles were analyzed for chemical composition in regions 1-6. The analyses indicate that the sub-spherical particles primarily consist of uranium (~75 %, consistent with the overall uranium concentration of the CUP-2 standard), with oxygen making up the bulk of the remaining chemistry. These findings are consistent with a U_3O_8 material. Other elements identified in the sub-spherical particles include aluminum, sulfur and calcium. The dark, platy particles were also analyzed (region 1); these particles seem to lack uranium. Rather, they contain abundant calcium, sulfur, and oxygen, indicating that these must be some kind of other, discrete crystalline phase. Because of the carbon substrate, most of the EDX spectra of the particles in the sample showed carbon peaks and those were excluded for the quantification measurements. It should also be noted here that the quantification numbers provided by SEM-EDX analysis are semi-quantitative since the measurements were performed with the standard instrument calibration, instead of a similar geometry and matrix-matched reference sample.



Spectrum	U	O	Al	S	N	Si	Ca	Fe
1	25.36	32.77	**2.88**	**12.65**		1.84	**24.5**	
2	70.22	25.51	0.61	2.63		0.52		**0.51**
3	75.21	19.77	0.96	4.05				
4	78.93	16.93	0.56	1.53	2.04			
5	86.62	11.79	0.92	0.68				
6	92.07	4.24	**3.68**					

Figure 7. Energy-dispersive X-ray analysis and corresponding backscatter image of CUP-2 particles.

SAMPLE PREPARATION FOR DESTRUCTIVE ANALYSES

Approximately 1.8 g of CUP-2 powder was separated from the stock bottle for destructive analyses. Prior to dissolution, the sample was crushed using an agate mortar and pestle, pre-cleaned with clean SiO_2 sand and pre-contaminated with a small amount of the sample. The sample is crushed until it appeared homogeneous in terms of particle size and color. The crushed and homogenized powder was then stored in a clean glass vial for sub-sampling. The aliquots were divided as follows:

-50 mg for X-ray diffraction
-250 mg for X-ray fluorescence
-600 mg for microwave digestion (for ICP-MS and Davies-Gray analyses)
-100 mg for uranium isotopic composition analysis

In order to minimize the blank contribution introduced during dissolution, all acids used in the following procedures are Seastar BASELINE grade, in which most elements have a concentration of <10 parts per trillion. All acid dissolutions are prepared using ultra-trace grade water with a resistivity of 18.2 MΩ or better. In most cases, bulk U materials will dissolve readily in 8 M HNO_3. However, to stabilize the high field strength elements, such as Zr and Hf, the addition of a small amount of HF is necessary. However, it is important to note that the addition of HF to a dissolved sample containing silicon will result in the loss, and inability to quantify, Si in the sample.

The microwave digestion system used in this procedure is a CEM MARS microwave equipped with EasyPrep vessels. A microwave batch consists of 11 regular vessels and a control vessel. The control vessel is monitored for both temperature and pressure, and should contain the sample with the most material in order to assess whether or not the non-monitored samples were to self-vent.

The weighed sample is added to a pre-cleaned microwave vessel followed by 1 mL of 18.2 MΩ water to rinse the sample to the bottom of the vessel. Next, 9 mL of a concentrated HNO_3 + 0.09 M HF solution is added to each vessel. Samples are allowed to sit for 15 minutes to complete any initial reaction related to the dissolution of organic material, and are then loaded into the microwave. The temperature control program ramps the vessels to a maximum of 180C over 20 mins with a hold time of 15 mins. When the program is complete, the vessels are allowed to cool to room temperature prior to opening. Samples are visibly inspected to ensure complete dissolution. On rare occasions, a sample is not completely dissolved. In this case, an additional amount of HF is added to the vessel, and the sample is processed through an additional microwave session.

Upon completion of the microwave procedure, the dissolved samples are quantitatively transferred to a set of pre-cleaned, pre-weighed 50 mL "trace metal free" centrifuge tubes and diluted with 18.2 MΩ water so that the total solution volume is approximately 50 mL. The sample is then thoroughly shaken to ensure a homogeneous solution is produced, and the final mass of the solution + centrifuge tube is weighed and recorded. The final acid concentration is approximately 2.9 M HNO_3 and 0.016 M HF.

X-RAY DIFFRACTION

All samples are analyzed as received (powdered) and are scanned from 10 to 70° 2. The samples are either top loaded into a Poly methyl methacrylate (PMMA) sample holder, or top loaded as a slurry onto a zero background silicon wafer PMMA sample holder. The step scan parameters are 0.02° step and 2 second counting time per step. The usual slit parameters are a 12mm variable divergence slit and a 0.499° antiscatter slit.

All samples are X-rayed with Ni-filter Cu radiation from a sealed Cu tube operated at 40kV and 40mA. Phases in the unknown samples are identified by comparison of observed peaks to those in the International Centre for Diffraction Data (ICDD PDF2009) powder diffraction database.

Method of validation with sintered Al_2O_3: X-ray reference material (Bruker supplied Al_2O_3) is analyzed with all unknowns to ensure goniometer alignment. The sintered Al_2O_3 is scanned from 30 to 40° to encompass the strongest Al_2O_3 peaks. The standard scan is analyzed for peak shifts from known values. A data file of all Al_2O_3 standard scans is kept for validation records.

The X-ray instrument housed in B151 R1143N is a Bruker AXS D8 ADVANCE X-ray diffractometer (serial number 4202/842-071200), equipped with a LynxEye 1-dimentional linear Si strip detector. DIFFRAC plus XRD Commander, version 2.6.1 is used to operate the goniometer, and DIFFRACplus Evaluation package, version 15,0,0,0 Release 2009 (EVA) is the Bruker supplied data analysis software.

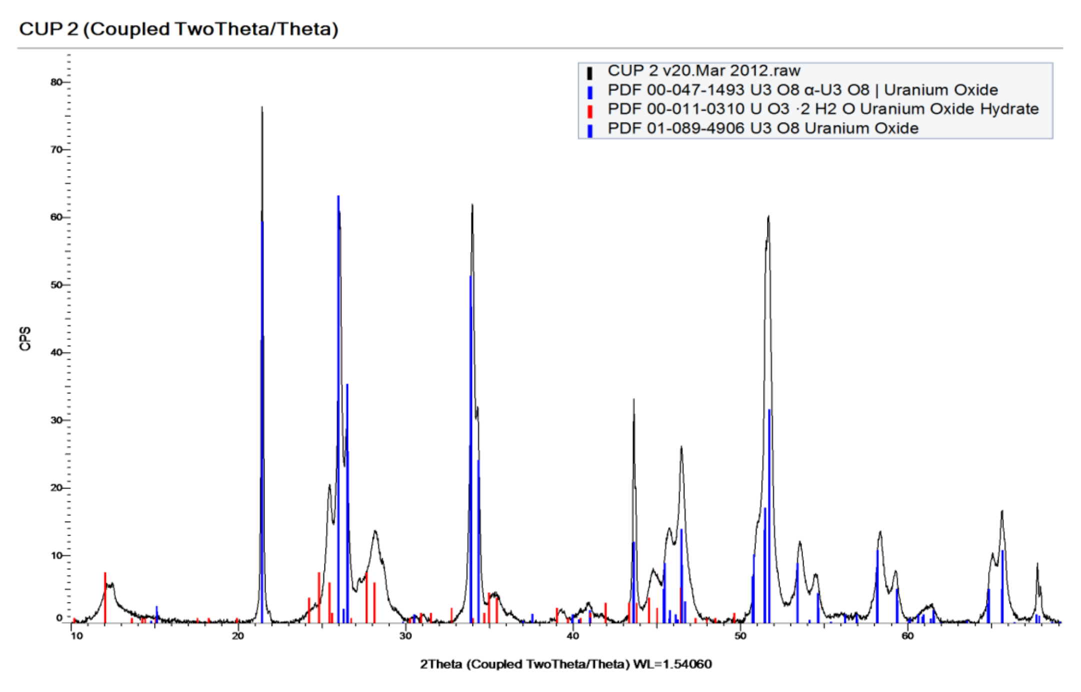


Figure 8. X-ray diffraction spectrum (black spectrum) of one aliquot of CUP-2, with peaks identified using the ICDD PDF2009 powder diffraction database. This analysis of CUP-2 is consistent with U_3O_8 (blue lines) and $UO_3 \bullet 2H_2O$ (red lines).

Analysis of the CUP-2 sample was performed on six separate homogenized aliquots, and one aliquot that had been dried in an oven for several hours, to remove water. The results of one analysis can be found in Figure 8 and Table 6. Spectra produced by the analysis of "wet" homogenized sample were consistent with the presence of U_3O_8 as the major phase present, and with $UO_3 \bullet 2H_2O$ being a minor phase. Although the specific peak heights of replicate analyses of CUP-2 tend to vary, all analyses are consistent with these findings. In the case of the dried sample, the most definitive peak for the $UO_3 \bullet 2H_2O$ phase disappears (light green line in Figure 9), indicating that the presence of this phase is due to the presence of moisture in the sample.

Angle	d Value	Net Intensity	Gross Intensity	Rel. Intensity
12.20751	7.24448	2161	6201	7.43%
12.44340	7.10767	2134	6191	7.34%
21.41964	4.14507	29093	32905	100.00%
25.44538	3.49767	7869	12077	27.05%
26.05424	3.41729	23194	27466	79.72%
26.42435	3.37026	10543	14847	36.24%
27.24827	3.27020	2568	6926	8.83%
28.17387	3.16483	5262	9651	18.09%
28.55037	3.12395	3554	7946	12.22%
31.04936	2.87798	525	4811	1.81%
33.98461	2.63582	23726	27890	81.55%
34.23736	2.61694	11811	15970	40.60%
35.23855	2.54484	1668	5783	5.73%
35.34814	2.53720	1516	5624	5.21%
39.27564	2.29206	755	4749	2.60%
40.91391	2.20398	1112	5311	3.82%
43.65121	2.07191	10693	15244	36.76%
44.80348	2.02126	3009	7785	10.34%
45.75084	1.98159	5375	10300	18.48%
46.48122	1.95214	9965	14982	34.25%
51.07398	1.78685	5759	11307	19.79%
51.63056	1.76889	22852	28473	78.55%
53.55476	1.70978	4604	10392	15.83%
54.49109	1.68260	2800	8619	9.62%
58.32575	1.58077	5193	10810	17.85%
59.24715	1.55837	2969	8458	10.20%
61.37126	1.50942	981	6057	3.37%
65.07230	1.43223	3912	8961	13.45%
65.62447	1.42151	6339	11475	21.79%
67.75581	1.38189	3102	8473	10.66%
67.89024	1.37948	1672	7053	5.75%

Table 6. Results of X-ray diffraction analysis of a single aliquot of CUP-2.

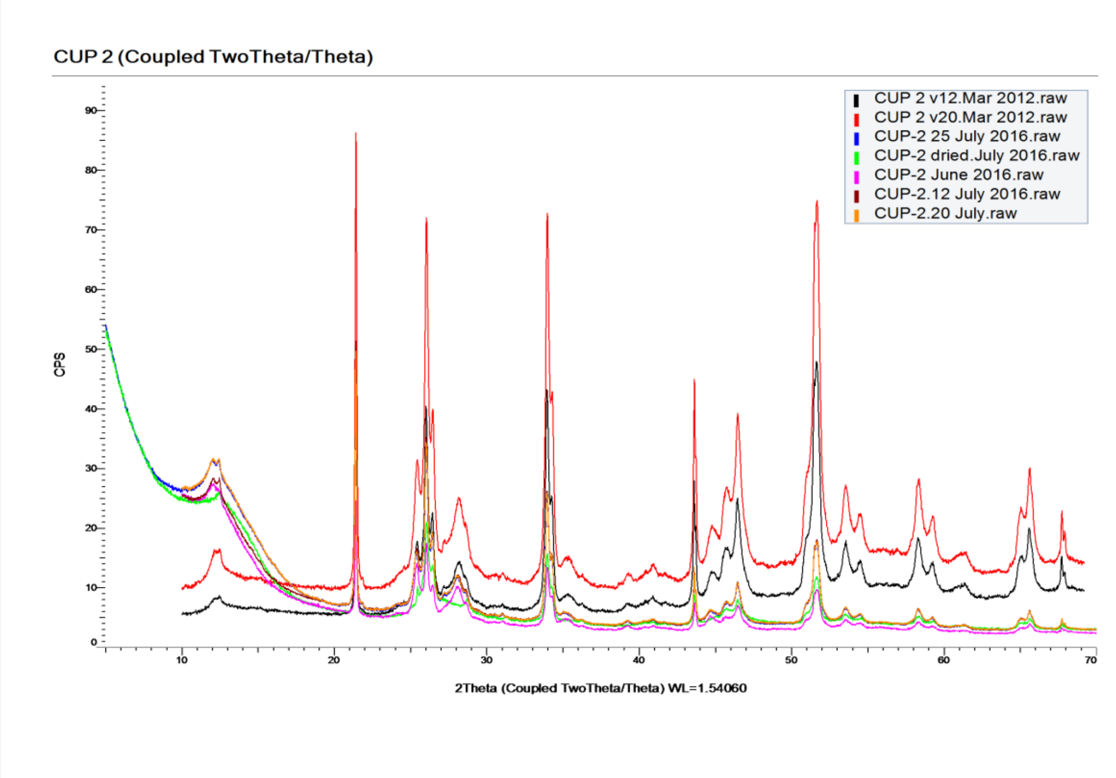


Figure 9. X-ray diffraction spectra of 6 homogenized and one homogenized + dried aliquots of CUP-2.

X-RAY FLUORESCENCE

X-ray fluorescence (XRF) spectrometry was performed to determine the concentrations of trace element impurities in the CUP-2 UOC powder. Measurements were performed using a Bruker S8 wavelength-dispersive XRF at LLNL. Analyses were performed by loading approximately 250-500 mg of loose powder into sample cups supported by 12 μm thick polypropylene films across 8 mm orifices, and capped using microporous film, which prevents the reduced-pressure condition of the sample chamber from rupturing the sample cup. Data were collected using the Bruker QuantExpress analytical program. The instrument calibration was performed by measuring a set of standard silicate glass discs (Breitländer GmbH), certified for a suite of major and trace elements, under the same operating conditions as the samples. Even though all elements from Na to U were analyzed; only elements measured above detection limits are reported (<DL, below detection limit). Detection limits were typically on the order of 50-200 μg/g. Totals were typically below 100 %; results were normalized to 100 %. Iron is reported as Fe^{2+}. The results of the analyses can be found in Table 7.

Elements were measured as oxides (not as elements) in semi-quantitative mode using a helium atmosphere. The set of standard glass discs were analyzed at the start of each analytical work day to assess precision and accuracy of the semi-quantitative calibration curve. X-ray tube voltage and current varied depending on the element being measured at any given time. All elements $Z \geq 11$ (Na to U) were measured.

After analysis, qualitative XRF spectra were screened to assess accuracy of the peak search algorithm. Quantitative results were calculated based on measured net intensities using the EVAL2 software package. The relationship between net intensities and concentrations were determined using the built-in semi- quantitative calibration curve. Results deriving from misidentified peaks were discarded. Elemental concentrations were calculated and normalized to 100 %. Oxygen concentration was calculated by assuming typical stoichiometric proportions in geologic samples.

Lower limit of detection is calculated using the formula

$$LLD=3/m\sqrt{(I\_b/T\_b)}$$

where m = sensitivity of analyte in kcps/mass %, Ib = background intensity of analyte in kcps, and Tb = counting time in seconds. The detection limits of Cu (150 ppm), Fe (100 ppm), Cr (50 ppm), and Ni (50 ppm) are constant due to the presence of these elements in the physical components of the instrument.

μg/g	CUP-2 (1)	+/- (2σ)	CUP-2 (2)	+/- (2σ)	CUP-2 (3)	+/- (2σ)	CUP-2 (4)	+/- (2σ)	CUP-2 (5)	+/- (2σ)
Ca	7600	500	10300	500	10300	500	11700	500	10200	500
S	8300	300	8500	300	8800	300	9500	300	8200	300
Fe	5200	300	4600	200	5200	200	4180	190	4600	200
Th	3200	200	2800	170	2900	180	2500	170	2700	180
Si	1300	300	1800	200	1700	200	1400	200	1700	200
Mg	1300	300	<DL	-	<DL	-	<DL	-	<DL	-
Ti	1200	200	<DL	-	<DL	-	<DL	-	<DL	-
V	1200	200	750	160	<DL	-	1100	170	790	170
As	580	70	<DL	-	480	50	430	50	440	50
Al	<DL	-	2300	300	1600	300	1200	300	<DL	-
Na	<DL	-	<DL	-	<DL	-	3500	1000	<DL	-
K	<DL	-	<DL	-	2700	200	2400	200	2900	300
Cl	<DL	-	<DL	-	630	140	650	140	530	140

Table 7. Results of five replicate analyses of CUP-2 UOC samples by XRF. All data in μg/g sample.

The results of the XRF analyses were broadly consistent with the certified concentrations of trace elements in CUP-2. The XRF technique was able to quantify the more abundant trace elements, such as Ca, S, and Fe, but was unable to quantify several trace elements of lower abundance, such as B. Na is difficult to quantify by XRF due to the low fluorescence yield of that element. Zr and Mo cannot be quantified by XRF due to the presence of U spectral interferences on these lines.

URANIUM ASSAY BY ELECTROCHEMICAL TITRATION

Uranium concentration measurements were performed for three separate aliquots of the CUP-2 uranium ore concentrate standard. Samples were dissolved following the methods utilized for ICP-MS measurements. Analyses were performed using a Metrohm auto-titration system. The system utilizes a set of five burets, dedicated for each reagent.

Reagents were prepared using the following procedures:

- Potassium dichromate – a stock solution of 0.045 M $K_2Cr_2O_7$ was prepared by dissolving 2 g crystalline $K_2Cr_2O_7$ in 100 g H_2O. A 10:1 dilution of this solution was then prepared and used in the analytical procedure.
- Sulfamic acid – Approximately 146 g of crystalline sulfamic acid was dissolved in 1 L of H_2O, and thoroughly mixed.
- Ferrous sulfate heptahydrate – A stock solution of $FeSO_4 \cdot 7H_2O$ was prepared by adding approximately 70 g of crystalline $FeSO_4 \cdot 7H_2O$ to a solution of 25 mL H_2SO_4 and 225 mL H_2O, and thoroughly mixed. A 60 mL aliquot of this solution was then added to 500 mL of H_3PO_4, and thoroughly mixed. This mixture was used in the analytical procedure.
- Ammonium molybdate with sulfamic acid – Approximately 500 mL of concentrated HNO_3 was added to approximately 400 mL of H_2O. 100 mL of the sulfamic acid solution was then added to this mixture. Approximately 4 g of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ was then added, and thoroughly mixed.
- Vanadyl sulfate dihydrate – Approximately 50 mL of H_2SO_4 was added to 950 mL of H_2O, thoroughly mixed, and cooled to room temperature. Approximately 1 g of crystalline $VOSO_4 \cdot 2H_2O$ was then added to the solution, and thoroughly mixed.

All solutions were transferred to their respective burets. Aliquots of the digested CUP-2 material were transferred to Pyrex beakers. Masses were obtained for each solution to the fourth decimal place. Enough of the CUP-2 solutions was transferred to deliver 30-70 mg of total U for analysis. After transferring the solutions to the beakers, 1 mL 1 M H_2SO_4 was added to each beaker, and then dried using a hot plate set to ~315°C. Upon dryness, the samples were allowed to cool, and then dissolved using 2 mL 1 M HNO3 for analysis.

The titration is performed using the following procedure:
-2.5 mL of sulfamic acid and 15 mL of ferrous sulfate heptahydrate were added to the sample, and the solution was stirred for 25 seconds
-3.4 mL of ammonium molybdate with sulfamic acid was added to the sample, and stirred for 4 minutes. The solution initially turned black, and then began to form bubbles, after about 30 seconds, the solution turned light blue, and began to exsolve the bubbles that formed.
-34 mL of vanadyl sulfate dihydrate was added, and the solution was stirred indefinitely
-While stirring, the potassium dichromate was added in small increments. After the addition of each increment, the electrical potential of the solution was observed, as measured by a platinum electrode. Electrical potential initially rose slowly, but increased after the addition of several mL, at a voltage of approximately 550 mV. Smaller increments of potassium dichromate were then added at this point. The endpoint of the titration was achieved when the solution potential reached 650 mV. At this point, the mass of potassium dichromate titrant utilized to achieve this potential was recorded.
-The electrode and all solution tubing were thoroughly rinsed prior to the next set of analyses.

The titration factor of the potassium dichromate titrant is a function of its concentration, and is very sensitive to slight differences in concentration. In order to determine the titration factor (mg U/g $K_2Cr_2O_7$), a uranium concentration standard, made by dissolving a uranium standard of natural uranium isotopic composition, and confirming the concentration of uranium in this solution using replicate measurements of isotope dilution mass spectrometry. The uranium titration factor was calculated using the equation:

T= (C_U)(G)(W)

where T is the uranium titration factor, CU is the concentration of the standard uranium solution (determined using another independent method), G is the mass of the aliquot of the standard uranium solution, and W is the mass of the aliquot of the potassium dichromate titrant used to achieve a potential of 650 mV during the titration of the standard uranium solution. The potassium dichromate titrant should be calibrated each time a new batch is prepared.

Sample ID	**wt % U**	**Uncert. (k = 2)**
CUP-2-A	73.49	0.17
CUP-2-B	73.00	0.17
CUP-2-C	73.15	0.17

Table 8. Results of Davies-Gray uranium concentration analyses.

The results of three replicate analyses of CUP-2 are presented in Table 8. Concentrations vary from 73.00 ± 0.17 to 73.49 ± 0.17 wt. % U. Two of the three analyses (CUP-2-B and CUP-2-C) overlap within uncertainty, and CUP-2-A barely overlaps within uncertainty with CUP-2-C (CUP-2-A is higher outside of uncertainty than CUP-2-B). All three of these measurements are lower than indicated by the CUP-2 certificate. However, according to the certificate, the certified uranium CUP-2 concentration is for a "dry-weight" basis, whereas these measurements were conducted on an "as-received" basis. This may explain the disparity between the certified and measured values.

INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY (ICP-MS)

CUP-2 and its associated process blanks were analyzed on a Thermo Scientific iCAP-Q Quadrupole ICP-MS. The procedure employed for trace element analysis of bulk uranium by ICP-MS includes three separate digestions and analyses, each made on a separate day. Therefore, each aliquot is treated as an independent measurement. By following this procedure, it is possible to produce a representative sample population that incorporates day to day analytical uncertainty.. Samples are dissolved and analyzed on the same day, whenever possible, to ensure the stability of the trace elements in the prepared solutions.

Due to the high concentration of uranium in uranium ore concentrate, and the necessity of running U-rich solutions at relatively high concentrations in order to ensure that trace elements are present in quantifiable concentrations, uranium-rich matrices are a challenge in terms of ICP-MS signal suppression and space charge effects. Tuning the ICP-MS with a U-rich matrix optimizes the analytical conditions. Internal standard correction is also employed to correct for sensitivity drift and the effect of uranium suppression overall. For this analysis In and Rh were added to each standard and sample at a concentration of 1 ppb.

A daily mass calibration is not necessary; however, the mass position and width of ^{6}Li, ^{115}In, and ^{238}U was assessed in the daily performance report to ensure that the peaks were within their analytical windows. In addition, a cross-calibration of the SEM detector was performed, to minimize the offset between the detector's pulse-counting and analog modes.

The first step in the trace element analysis is a semi-quantitative analysis of the unknown samples. This analysis serves 2 purposes: the first is to determine the U concentration in order to identify the appropriate dilution to achieve a 100 µg/g U solution. The second is to identify any elements that may fall outside of the external calibration in the 100 µg/g U solution. The unknown samples are gravimetrically diluted to approximately 1-2 µg/g U solution. The unknown is quantified for uranium using a one point U calibration curve and for trace elements using a separate 1 point multi-element calibration curve. For CUP-2, only Th fell outside of our calibration range. The measured intensities were ten times greater than the highest calibration standard and therefore a special dilution was made.

All sample and process blank stock solutions were diluted gravimetrically with a 1ppb internal standard solution of In and Rh in 2% nitric acid and 0.005M hydrofluoric acid for a final volume of 10ml.

Trace element concentrations are determined by analyzing the sample solutions, and calculating concentrations by using the results of a set of calibration solutions, both matrix-matched (100 µg/g U) and non-matrix-matched. The external calibration curve consists of 8 solutions prepared from NIST traceable custom commercial mixtures that cover a 104 range in concentration, for 57 elements. Elements present in the calibration curve include Li, B, Be, Na, Mg, Al, P, S, K, Ca, Sc,Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Rb, Sr, Y, Zr, Nb, Mo, Ag, Cd, Sn, Sb, Te, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, W, Re, Tl, Pb, Bi and Th. The matrix-matched standards are spiked with an approximately 20,000 µg/g U solution made from NBL 112A, a U metal of natural isotopic composition, to achieve a 100 µg/g U solution. Concentrations for almost all of the reported elements were calculated using the results of the matrix-matched calibration curve. The non-matrix-matched calibration curve was used for the process blanks and for the larger (500x) dilution used to quantify Th in CUP-2.

In general, selection of isotopes for each element of interest was based on its natural abundance and the lack of a direct isobaric interference. Whenever possible, more than one isotope was measured for each element, providing multiple options in case of unforeseen polyatomic interferences. In the case of U-rich matrices such as UOC, samples may contain both radiogenic and common Pb. Due to possible differences in Pb isotopic abundance, total Pb was accounted for by measuring all isotopes of Pb (^{204}Pb, ^{206}Pb, ^{207}Pb, and ^{208}Pb) Furthermore, while the production of ^{230}Th from the 238U decay chain is small in natural uranium, total Th measurement included both ^{230}Th and ^{232}Th.

Integration times were optimized for each element or group of elements, depending on expected concentrations. For example, the rare earth elements were assigned longer integration times to improve counting statistics and obtain a more precise measurement.

Direct isobaric interferences, such as ^{115}Sn on the internal standard element ^{115}In, were corrected for by monitoring a different isotope of Sn (^{120}Sn), and correcting for the interference based on the natural Sn isotopic abundance. Some polyatomic interferences are corrected manually, such as ^{95}Mo^{16}O on ^{111}Cd, and light REE oxides on heavy REEs. The magnitude of the correction was determined based on the oxide production efficiency for each element on the day of analysis by measuring single element solutions of the isotope producing the interference. The correction is made based on the signal intensity of a single isotope (e.g. ^{95}Mo) relative to potential polyatomic species (e.g. ^{95}Mo^{16}O), and from this determination, a correction is made to the mass of interest (e.g. ^{111}Cd).

Both U and U molecular polyatomic compounds for doubly-charged species can interfere with elements of interest. For example, the $^{238}U^{++}$ interference on ^{119}Sn poses not only a challenge to quantifying Sn, but also for the isobaric interference correction on ^{115}In. Therefore we used ^{120}Sn to quantify Sn to avoid this interference. Also, sub-ppb concentrations of La can be difficult to measure in a 100 ppm U solution, due to the $^{238}U^{40}Ar^{++}$ interference. The current procedure is to subtract the $^{238}U^{40}Ar^{++}$ contribution from the unknown using the matrix-matched blank solution.

The analytical sequence was compiled in the following order: instrument blanks, calibration standards, QA/QC standards, process blanks, and unknowns. Whenever possible, unknowns should be analyzed in order of increasing concentration, to reduce sample-to-sample memory effects. The ICP-MS sample introduction system is rinsed out after each analysis using a solution of 5 % HNO_3 + 0.01 M HF. Instrumental rinse times should be of sufficient length to ensure that the signal intensities of the more sorbent elements are able to return to background levels. In some cases, multi-step rinse cycles are recommended.

Data on the UOC samples was collected as raw intensities (counts per second) with a 1σ standard deviation. The measured intensities were then corrected for any isobaric and/or polyatomic interferences, then normalized for in-run sensitivity drift and matrix suppression that occurred during the course of the run. Instrumental drift is corrected for using the measured intensities of the internal standard elements; in this case, ^{103}Rh was employed to correct all of the other measured elements. These corrected intensities were then blank-corrected, using the average intensities of three blank solutions measured at the start of each run.

After performing interference corrections, sensitivity drift corrections, and blank corrections, the intensities were converted to concentrations using the measured intensities of the multi-element standard calibration solutions. A calibration curve for each element is built using the measured signal intensities and known concentrations of each isotope in each calibration solution. The choice of whether to use the matrix-matched or non-matrix-matched calibration curve is made for each element of interest. Final solution concentrations were calculated in units of µg/g solution using the dilution factor of each solution. These values were then calculated to µg/g sample based on the final weight of each solution after digestion, and the mass of the sample dissolved. Finally, the 3 replicate analyses (n=3) were averaged for a final reported value (Table 9 and Table 10).

Uncertainties were propagated at each stage of the data reduction. The final uncertainty was multiplied by 2 for 2sigma uncertainty. In addition, the 95% confidence interval was determined for the the 3 replicates by the following equation: t*stdev + (n ½). Where t = t-statistic at 95% confidence limit at (n-1) degrees of freedom = 4.33; stdev is the standard deviation of the replicate measurements and n = number of replicate samples analyzed =3. The final uncertainty represents the larger of two values, either the propagated uncertainty or the 95% CI of the 3 replicates.

The detection limits were calculated as a detection decision Lc, which is defined as a 1% rejection or 99% confidence interval. To calculate, the standard deviation of the three process blanks was multiplied by the t-statistic for 99% confidence interval at n-1 degrees of freedom: Lc = t*σB where t = 9.925.

Element	**November 2015 Quad CUP-2 (n=3) ug/g sample**	**95%CI**	**Reference Values CUP-2 ug/g sample**	**%R of AVG**
B	51.7	5.4	51	101%
Na	4280	170	4590	93%
Mg	2293	42	2290	100%
K	1090	120	1100	99%
Ca	6090	330	6200	98%
Ti	162.8	4.0	190	86%
V	677	21	660	103%
Fe	3330	66	3110	107%
Ni	25.3	2.2	29	87%
As	332	40	350	95%
Zr	424.4	4.2	440	96%
Mo	754	24	690	109%

Table 9. Results of ICP-MS analyses for the elements characterized by the certificate of analysis.

Trace element concentrations were compared to the recommended values provided by the CUP-2 certificate (see Table D). Measured concentrations for the elements B, Na, Mg, K, Ca, Ti, V, Fe, Ni, As, Zr, and Mo generally agreed favorably with the recommended values (note that the certificate values do not have an uncertainty associated with them). Most of the measured elements were within 10 % of the recommended values, typically overlapping within uncertainty. Only two elements (Ti and Ni) were greater than 10 % outside of the recommended values, and did not overlap within uncertainty.

Element	CUP-2 (n=3)	
	ug/g sample	95%CI
Li	N.M.	-
Be	0.258	0.070
Al	2380	180
P	N.M.	-
S	N.M.	-
Sc	4.84	0.20
Cr	15.07	0.33
Mn	97.2	1.5
Co	2.283	0.056
Cu	22.15	0.66
Zn	32.69	0.84
Ga	0.519	0.035
Rb	7.74	0.41
Sr	53.80	0.39
Y	75.2	1.2
Nb	0.78	0.68
Ag	0.298	0.016
Cd	0.206	0.074
Sn	3.53	0.48
Sb	0.147	0.061
Cs	0.996	0.027
Ba	110.5	1.6
La	19.35	0.45
Ce	39.57	0.65
Pr	4.919	0.078
Nd	19.83	0.29
Sm	9.27	0.10
Eu	0.763	0.016
Gd	14.69	0.21
Tb	3.014	0.055
Dy	18.64	0.29
Ho	3.311	0.047
Er	8.67	0.15
Tm	1.156	0.017
Yb	6.824	0.075
Lu	0.825	0.014
Hf	0.3744	0.0074
Ta	0.0599	0.0041
W	14.28	0.37
Tl	3.576	0.059
Pb	283	29
Bi	1.481	0.039
Th	1800	270

Table 10. Results of ICP-MS analyses for the elements not characterized by the certificate of analysis. N.M., not measured.

ISOTOPE RATIO MASS SPECTROMETRY

Chemical purification for uranium

In order to perform accurate and precise uranium isotopic analyses, it is critical to separate and purify the uranium in the CUP-2 sample. The most commonly-employed method, and the method we employed here, is chemical separation by ion exchange chromatography. In this method, a series of chemical separation steps are employed utilizing ion exchange resins of with different types (e.g. HCl, HNO_3) and concentrations of acids. Depending on the resin, acid, and acid concentration, certain elements present in the sample will sorb to the resin, while others will not sorb. Using this method, it is possible to quantitatively recover uranium from the sample, while removing other interfering elements. As some UOC samples contain considerable concentrations of impurities, as does the CUP-2 standard, one or more chemical separation steps may be required, depending on the sample.

Aliquots of 100 mg were dissolved in 3 mL 10.5 M HCl and 50 µL concentrated HNO_3, in clean and dry Teflon beakers. Typically, samples will dissolve on a hot plate when heated overnight in this solution. It is critical to ensure that the sample is completely dissolved at this point. The sample is then dried down overnight, and dissolved in ~1 mL 10.5 M HCl. Columns, containing the anion exchange resin AG-1 X8 (Bio-Rad), 100-200 mesh grain size, are conditioned with 10.5 M HCl. The column is then loaded with the sample, and rinsed with additional 10.5 M HCl, removing most of the Pb, REEs, alkaline earth metals, and other elements (uranium sorbs to the resin in 10.5 M HCl). The uranium is then recovered by washing the resin repeatedly with 0.1 M HCl, and collecting the eluate in a clean Teflon beaker. The eluate is then dried down on a hot plate overnight.

An additional purification step is necessary to achieve sufficient purity for uranium isotopic analysis by ICP-MS. After achieving dryness, the eluate from the previous step is dissolved in ~1 mL 4 M HNO_3. Columns, containing the ion exchange resin U-TEVA (Eichrom Technologies) are conditioned with 4 M HNO_3. The column is then loaded with the sample, and rinsed with additional 4 M HNO_3, removing the bulk of the remaining matrix elements, while uranium remains sorbed to the resin. Uranium is then eluted from the resin by repeatedly washing with 0.1 M HNO_3. The eluate is collected in a clean Teflon vial, and dried down overnight. After achieving dryness, the eluate is dissolved in 3 mL 2 % HNO3. At this point, the sample was ready for uranium isotopic analysis by ICP-MS. Additional dilutions should be made if one or more trace elements are not within the dynamic range of the instrument in this solution.

Multi-collector ICP-MS

The spiked and chemically-purified aliquots of uranium were measured using a Nu Plasma MC-ICP-MS. Samples were dissolved in 2 % HNO_3 for analysis. A static routine was used for Nu Plasma analyses, with ^{238}U and ^{235}U measured on Faraday detectors and ^{236}U, ^{234}U, and ^{233}U measured on ion counters. Prior to the analysis of each sample and standard, a blank measurement was performed on a solution of 2 % HNO_3; the measured blank intensities for each isotope were subtracted from the sample or standard measurement performed immediately after. Typically, these corrections are small, and have negligible effect on the corrected sample and standard intensities. A typical run consisted of 30 analytical cycles, with 10 s integration times. Mass bias correction factors and Faraday-ion counter gain factors were determined by measuring a solution of the New Brunswick Laboratory (NBL) U-010 uranium isotopic standard spiked with a calibrated concentration of ^{233}U. The ^{233}U-spiked U-010 standard was analyzed every three to four analyses of the CUP-2 sample. All calculations of uncertainty are performed following the guidance of Working Group 1 of the Joint Committee for Guides in Metrology (JCGM/WG1) as described in the document "Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement (JCGM 100 : 2008). Uranium isotopic compositions are reported as a combined standard uncertainty with a coverage factor of k = 2 (Table 11).

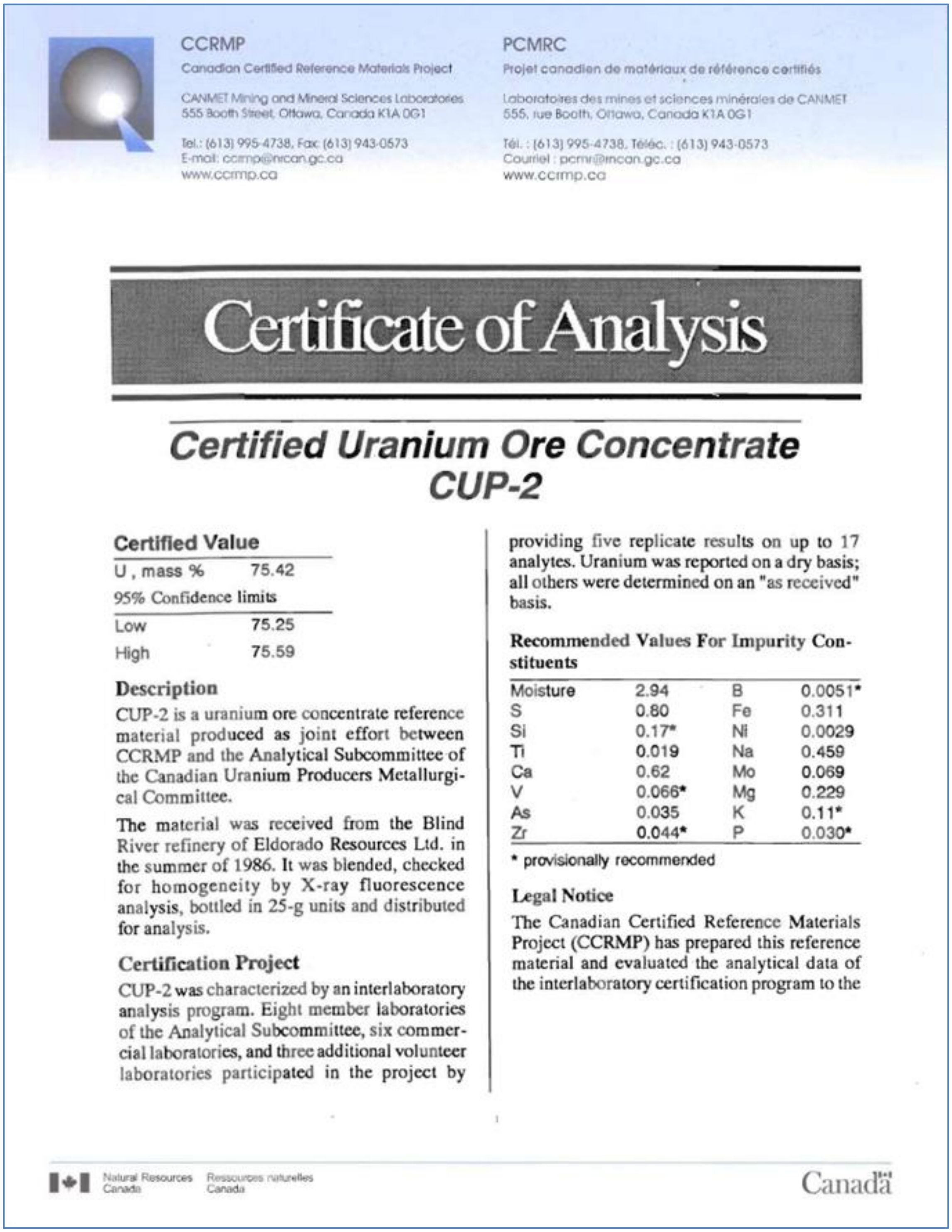
Analysis	Measured ratio	2 x CSU
$^{233}U/^{235}U$	0.0000001	0.0000012
$^{234}U/^{235}U$	0.00745	0.00003
$^{236}U/^{235}U$	0.000001	0.000006
$^{238}U/^{235}U$	137.86	0.14

Table 11. Results of MC-ICP-MS analyses.

The measured $^{238}U/^{235}U$ of the CUP-2 standard was 137.86 ± 0.14, within uncertainty of the composition of natural uranium (137.88). This is consistent with the results of both the non-destructive and destructive gamma spectrometry measurements. The measured

$^{234}U/^{235}U$ was 0.00745 ± 0.00003. When given as an activity ratio, the $(^{234}U)/(^{235}U)$ is 0.985 ± 0.004, slightly depleted in ^{234}U relative to its value in secular equilibrium, and is not unexpected in uranium ore concentrate samples. The uranium isotopes ^{233}U and ^{236}U, which do not occur naturally, were below the detection limit of this technique. These results indicate that the CUP-2 standard has a natural isotopic ratio, and does not appear to have been isotopically enriched or depleted in any way, and was not contaminated by a source of uranium with a non-natural isotopic composition. Furthermore, the lack of ^{233}U and ^{236}U above the instrumental detection limit indicates that this sample was not exposed to a neutron flux, which would have generated one or both of these isotopes in measurable concentrations.

Annex 1



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Certificate of Analysis

Certified Uranium Ore Concentrate CUP-2

Certified Value

U, mass %	75.42
95% Confidence limits	
Low	75.25
High	75.59

Description

CUP-2 is a uranium ore concentrate reference material produced as joint effort between CCRMP and the Analytical Subcommittee of the Canadian Uranium Producers Metallurgical Committee.

The material was received from the Blind River refinery of Eldorado Resources Ltd. in the summer of 1986. It was blended, checked for homogeneity by X-ray fluorescence analysis, bottled in 25-g units and distributed for analysis.

Certification Project

CUP-2 was characterized by an interlaboratory analysis program. Eight member laboratories of the Analytical Subcommittee, six commercial laboratories, and three additional volunteer laboratories participated in the project by providing five replicate results on up to 17 analytes. Uranium was reported on a dry basis; all others were determined on an "as received" basis.

Recommended Values For Impurity Constituents

Moisture	2.94	B	0.0051*
S	0.80	Fe	0.311
Si	0.17*	Ni	0.0029
Ti	0.019	Na	0.459
Ca	0.62	Mo	0.069
V	0.066*	Mg	0.229
As	0.035	K	0.11*
Zr	0.044*	P	0.030*

* provisionally recommended

Legal Notice

The Canadian Certified Reference Materials Project (CCRMP) has prepared this reference material and evaluated the analytical data of the interlaboratory certification program to the

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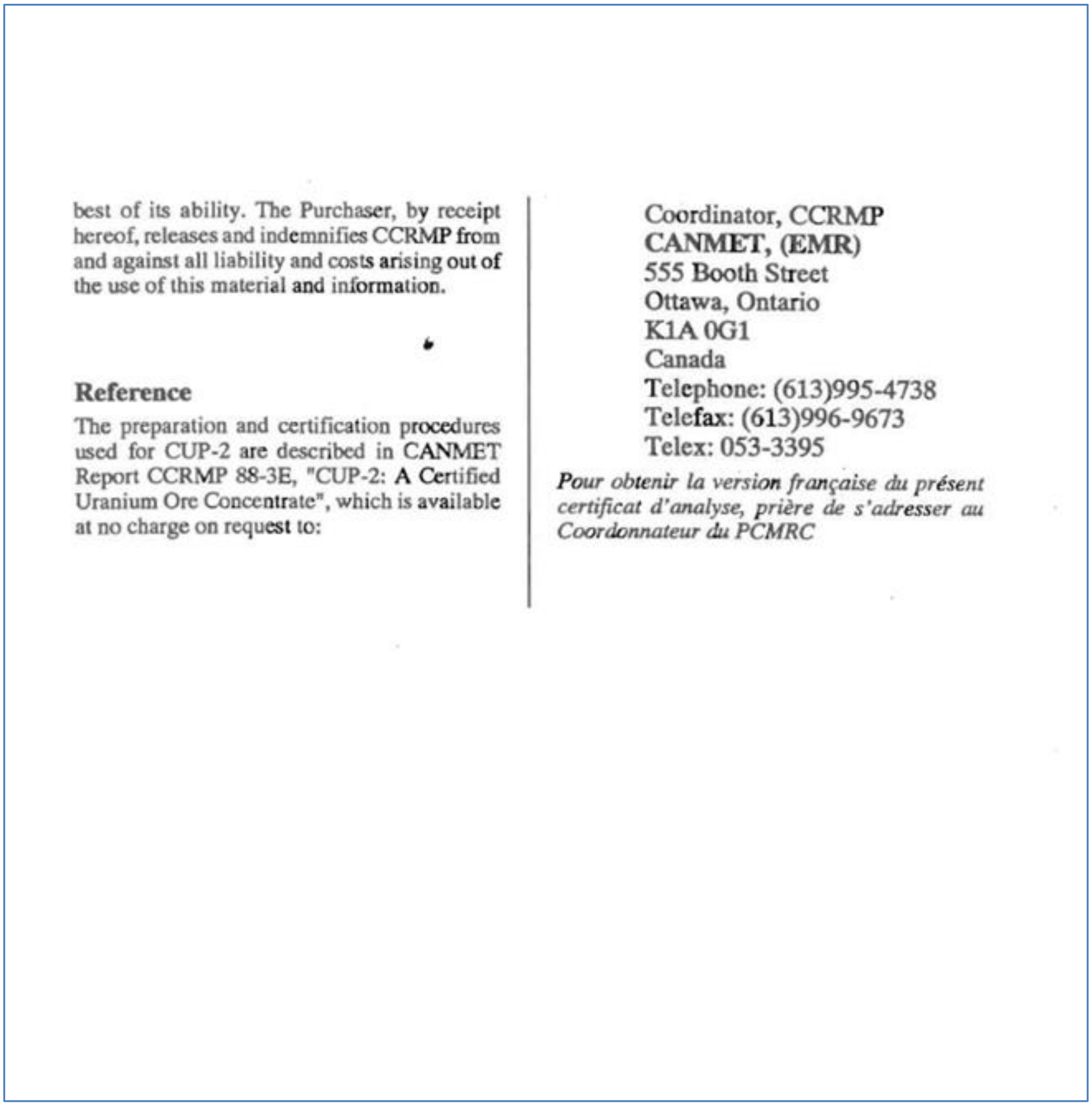

best of its ability. The Purchaser, by receipt hereof, releases and indemnifies CCRMP from and against all liability and costs arising out of the use of this material and information.

Reference

The preparation and certification procedures used for CUP-2 are described in CANMET Report CCRMP 88-3E, "CUP-2: A Certified Uranium Ore Concentrate", which is available at no charge on request to:

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Pour obtenir la version française du présent certificat d'analyse, prière de s'adresser au Coordonnateur du PCMRC